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USE OF A LASER FOR THE SPECTRAL ANALYSIS OF SEMICONDUCTOR MATERIALS

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16. Abstract

Conventional applications of lasers for emission spectroscopy involving direct recording of light pulses of an evapd. substance emitted from the sample under the action of the laser light (direct method) and use of the laser light for conversion of the substance to a vapor and feeding the vapors into the conventional source of emission such as arc, sparks, etc. (the so-called 2-stage excitation) were studied for use in the spectral anal. of semiconductors. The direct method has a high reproducibility (5-7%); the 2-stage excitation method, characterized by the same intensity as obtained with the conventional const. current arc, has better reproducibility than the direct method (15-20%). Both methods can be used for the anal. of samples without prior prepn. Advantages of these methods are the elimination of impurities picked up during trituration of the samples into powders and shortening of the analytical procedures.

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Modern science has various methods available for the control of the composition of substances. /59* Among these, spectral emission analysis is the most widely used. Convenient in many respects, it encounters a number of difficulties in analysis of pure and solid substances (diamond, ruby, carborundum). One of these difficulties involves the fact that during processing of samples (grinding to powder) contamination of them occurs. Moreover, sometimes a great deal of time and effort is expended in preparing the sample for analysis. The impossibility of retaining the sample in its original form is also a deficiency of the usual method of quantitative spectral analysis.

We attempted to get around these difficulties by using a laser (OKG [opticheskiy kvantovyy generator, laser]) in spectral analysis of semiconductor materials [1,2]. At the present time, lasers are used in emission spectroscopy for two purposes: first, it records directly the light pulses of an evaporated substance emitted from the sample under the effect of the laser beam (direct method); second, the laser beam is used for conversion of the substance into a vapor state and subsequently feeding the vapors into an ordinary spectral source, -- arc, spark, etc. (the so-called 2-stage method of spectra excitation). Both of these methods have their own advantages and disadvantages. The first method of direct fixation of the spark is attractive because of its simplicity, however the laser spectra obtained differ somewhat from the conventional, broadened and self-reversal large number of spectral lines; also, the presence of a background makes work with the spectrum difficult. Moreover, for a number of elements, the sensitivity of analysis is somewhat lower

*Numbers in the margin indicate pagination in the foreign text.

(approximately by an order of magnitude) than, for example, in a direct current arc. This, apparently, is explained by the short lifetime of the flare, the small quantity of evaporated substance (an order of 1 mg), and the low density of the vapors. At the same time, this method has good reproducibility (5-7%). /60

With the two-stage method, the character of the spectrum is determined by the spectral source selected. The sensitivity of determination is approximately the same as in the conventional direct current arc, but reproducibility is somewhat poorer than in the direct method (15-20%). With both methods, it is possible to conduct the analysis without preliminary preparation of the sample which permits avoiding contamination and saves time.

The experimental setup consisted of an Nd-glass laser (generation time $0.5 \cdot 10^{-3}$ s, energy ≈ 20 joules), a quartz PSP-22 spectrograph, and a low-voltage pulse discharge generator (total capactiy of the capacitors 8000 μF, voltage of the charge, 300 V). The spectra were photographed on "micro" plates (sensitivity 22 units GOST [Gosudarstvennyy obshchesoyuznyy standart, All-Union State Standard]).

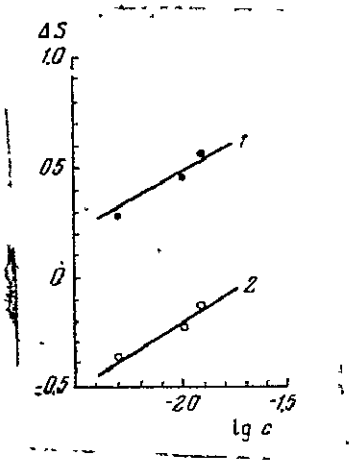
An attempt was made to use a laser for determining micro-impurities in pure semiconductor germanium and silicon. We expected that by accumulating sparks on one spectrogram that we would succeed in detecting < $1 \cdot 10^{-4}$% impurities. The results showed that with the two-stage method of excitation, even after 10 sparks, the background increased strongly which made a further accumulation of sparks senseless. Moreover, the lines of impurites which interested us (for example, Ga) were not visible. The accumulation of 30 sparks on the spectrum with the direct method of spectra excitation produced a similar result.

At the same time, the use of the laser for spectral deter- /61

mination of alloying elements (aluminum, boron, phosphorus) in other pure and solid semiconductor substances, in particular, in silicon carbide, was very successful.

The experiment showed that aluminum can be determined both by the direct and by the two-stage method up to a concentration of $1 \cdot 10^{-3}$ -- 10^{-4}%. Boron and phosphorus are determined only by the two-stage method where the secondary source is a low-voltage pulse charge between boron-less carbon electrodes.

A calibrated graph is reproduced in the drawing showing determination of boron in silicon carbide. Samples of silicon carbide with concentrations of boron in an interval of $1 \cdot 10^{-2}$ -- $5 \cdot 10^{-3}$% were used as the measurement standard (the boron was determined by an EPR [Elektronyy paramagneticheskiy rezonans, electron paramagnetic resonance] method). The reproducibility of analysis, found on the basis of 10 measurements amounted to 7-10% in our case.



A calibrated graph for determining boron in silicon carbide
1--B_I 2497[illegible]7--Si_I 2563.6; 2--B_I 2427.7--background

The peculiarity of the structure of silicon carbides alloyed with phosphorus, and also the absence of standards for phosphorus, made it impossible to conduct a quantitative analysis; a qualitative analysis along the λ=4943.5 Å and λ=4935.5 Å lines showed the presence in the samples of $1 \cdot 10^{-2}$% phosphorus.

The results obtained give one the basis for thinking that the use of lasers in quantitative spectral analysis of certain semiconductor materials will be very useful.

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